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EVALUATION OF THREE FUNGICIDAL TREATMENTS FOR WOOL FELT

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<p>— In order to compare the effectiveness of fungicidal treatments, wool felt sheet was treated with three fungicides and evaluated for resistance to leaching and microbial deterioration in soil burial.</p> <p>The dihydroxydichlorodiphenyl methane (G-4) treatment was nonprotective against microbial deterioration and the salicylanilide treatment was only marginally protective. Copper-8-quinolinolate was the only fungicide, in this study, that provided durable protection against biodeterioration in soil burial. The data suggest that copper-8-quinolinolate should be included as an alternative fungicide in Military Specification MIL-F-2312. In addition, performance testing (in this case, soil burial) should be included in military specifications wherever possible.</p>					
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PREFACE

This study was funded under the U.S. Army Natick Research, Development & Engineering Center 6.2 Program Element on the Prevention of Microbial Deterioration and/or Contamination of Military Material and Systems, Project No. 1L162723AH98, Work Unit CH001. All work was accomplished during FY85 and FY86.

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EVALUATION OF THREE FUNGICIDAL TREATMENTS FOR WOOL FELT

INTRODUCTION

Wool felt is composed of proteinaceous wool fiber which after manufacture retains residual oils, soaps, conditioning agents and debris. These constitute a susceptible material subject to both fungal and bacterial attack. To preserve and ensure aesthetic and functional values of wool in military goods, it is necessary that wool felt be protected from biodeterioration.

Wool felt is used by the Army in industrial and apparel applications. Military Specification MIL-F-2312E specifies mildew and moisture resistance requirements for felt - both hair and wool.¹ The mildew resistance requirement is for treatment with either 2,2' methylenebis-4-chlorophenol (G-4) or salicylanilide. Since these mildew resistance treatments are believed less effective than copper-8-quinolinolate (Cu-8) in protecting susceptible materials, a study was initiated, to evaluate the antimicrobial effectiveness of these preservatives relative to Cu-8, which is regarded as the choice fungicide for many military applications.

Preliminary microbial studies were performed on untreated wool felt samples.² These showed that samples of current production wool felt are highly susceptible to both fungal growth in plate tests and biodegradation in soil burial and therefore continue to require antimicrobial protection for military use.

This report contains laboratory data from felt treated with G-4, salicylanilide and Cu-8. These data serve to define the relative antimicrobial protection to be expected of these fungicides as preservatives for felt.

MATERIALS AND METHODS

Materials

All fungicides were evaluated on a white pressed felt sheet which was 3/16 inch (0.48 cm) thick. The felt sheet was manufactured by Commonwealth Felt Company of Northampton, MA, and conformed to Federal Specification C-F-206F Type I-mechanical-roll-felt, classification number 9R1 corresponding to S.A.E. felt number F-10.

Treatments

The treatments listed in Table 1 were diluted with tetrachloroethylene so as to meet mildew and moisture resistance requirements of Military Specification MIL-F-2312E, where applicable.

Table 1. Wool Felt Treatments

Treatment	MIL-F-2312E Fungicide Range on Felt, %	Fungicide Concentration of Bath, %
G-4 (Cuniphen 2173 ^a)	1.0-3.0	0.50
Cu-8 (Socci 3500WP ^a)	1.0-2.0 ^b	0.37
Salicylanilide + Cunipel 2498	0.5-1.5	0.50

^a Formulation includes moisture resistant finish.

^b Empirical treatment range selected on basis of past experience with Cu-8.

For Cu-8, felt was treated to deposit between 1% to 2% fungicide because that is an effective treatment level for the protection of most materials. Except for salicylanilide, all fungicides were proprietary formulations of Ventron Division of Morton Thiokol Inc., Danvers, MA. The Ventron formulations included wax and aluminum stearate to provide moisture resistance. Salicylanilide was a 99% pure compound manufactured by Aldrich Chemical Company, Inc., Milwaukee, WI. Salicylanilide was blended with sufficient Cunipel 2498 to meet the moisture resistance requirements of MIL-F-2312E. Cunipel 2498 composed of wax and aluminum stearate was also used to prepare the moisture-resistant felt control (containing no fungicide).

Application

After preliminary trials to optimize the fungicide concentration of each treating bath, samples 8 inches by 18 inches (20 cm x 46 cm) were soaked for 20 minutes in treating baths at fungicide concentrations listed in Table 1. The wet samples were wrung four times in an Atlas Laboratory Wringer (Atlas Electric Devices Co., Chicago, IL 60613) with the full 100 pound (45 kg) load applied to the rollers. After wringing, samples were allowed to air dry horizontally on aluminum foil. Pieces were turned over at about five minute intervals until dry to equalize fungicidal distribution throughout the material.

Methods

The methods used in this investigation are listed below.

1. Method 5762. Mildew Resistance of Textile Materials; Soil Burial Method³.
2. Method 5830. Leaching Resistance of Cloth; Standard Method.³
3. Method 5102. Strength and Elongation, Breaking of Woven Cloth; Cut Strip Method.³
4. Method 5502. Water Resistance of Cloth; Immersion Absorption Method.³
5. Method 2060. Copper-8-Quinolinolate Content of Textiles, Spectrophotometric Method.³
6. Method 2011. Dihydroxydichlorodiphenyl Methane Content, Colorimetric Method.³
7. Salicylanilide content was determined by Colorimetry.¹

RESULTS

Data from chemical analyses and moisture resistance tests following fungicidal treatment are compiled in Table 2. Felt samples were sectioned into thirds so that the top, bottom and interior layers could be analyzed separately. G-4 treated felt averaged 1.9% G-4 overall, with 1.8% G-4 in the interior section. Cu-8 treated felt averaged 1.5% Cu-8 overall, with 1.4% Cu-8 in the interior. Salicylanilide treated felt averaged 1.1% salicylanilide both overall and in the interior.

Table 2. Results of Chemical Analyses^a
and Moisture Resistance Tests^b following Fungicidal Treatment

Treatment	Analytical Concentration, %		Weight Increase after Immersion ^e
	Overall Means (SD) ^c	Interior Means (SD)	
None	d		90
Moisture Resistant (Cunipel 2498)	d		23
G-4 (Cuniphen 2713)	1.9 (0.6)	1.8 (0.7)	19
Cu-8 (Socci 3500WP)	1.5 (0.4)	1.4 (0.4)	40
Salicylanilide + Cunipel 2498	1.1 (0.3)	1.1 (0.3)	30

^aData based on six or more samples run in duplicate.

^bData based on multiple samples run in triplicate.

^cOne standard deviation.

^dNo fungicide applied and not analyzed.

^eNot to exceed 50% to be in compliance with MIL-F-2312E.

The untreated control increased 90% in weight after water immersion. Treated felt including the moisture resistant control ranged from 19% to 40% in water uptake.

After half of all specimens were leached, both the unleached and leached sets were subjected to soil burial. After soil exposure both sets of felt specimens were tested for loss of breaking strength (see Table 3). Figure 1 contains soil burial data from unleached specimens and Figure 2 from leached specimens. All sets of untreated controls, wax treated controls and G-4 treated felt specimens lost from 87% to 96% breaking strength after soil exposure for 2 weeks. Both sets of salicylanilide treated felt lost at least 50% strength by 4 weeks and 94% strength after soil exposure for 6 weeks. The unleached set of Cu-8 treated felt specimens lost no strength after soil exposure for 16 weeks but the leached set lost 31% strength after 16 weeks.

TABLE 3. Breaking Strength (N) = Results from Soil Burial Exposure of Sets of Five Replicate Specimens

Treatment	0	2	4	6	8	10	12
<u>None</u>							
Unleached	222 (9) ^a	9 (4)	- ^a	-	-	-	-
Leached	271 (9)	36 (4)	-	-	-	-	-
<u>Moisture Resistant</u>							
Unleached	263 (22)	13 (4)	-	-	-	-	-
Leached	258 (36)	22 (9)	-	-	-	-	-
<u>G-4</u>							
Unleached	254 (9)	9 (2)	-	-	-	-	-
Leached	218 (13)	27 (9)	-	-	-	-	-
<u>Cu-8</u>							
Unleached	258 (9)	271 (13)	276 (22)	263 (9)	258 (22)	294 (18)	263 (22)
Leached	285 (18)	289 (22)	267 (22)	289 (36)	258 (27)	245 (22)	196 (85)
<u>Salicylanilide + Cunipel 2498</u>							
Unleached	240 (22)	218 (36)	98 (9)	13 (4)	-	-	-
Leached	240 (22)	182 (36)	49 (13)	13 (4)	-	-	-

^aNewton (N) = pound-force x 4.45.

^bOne standard deviation.

^cToo weak for testing.

MR=MOISTURE RESISTANT CONTROL

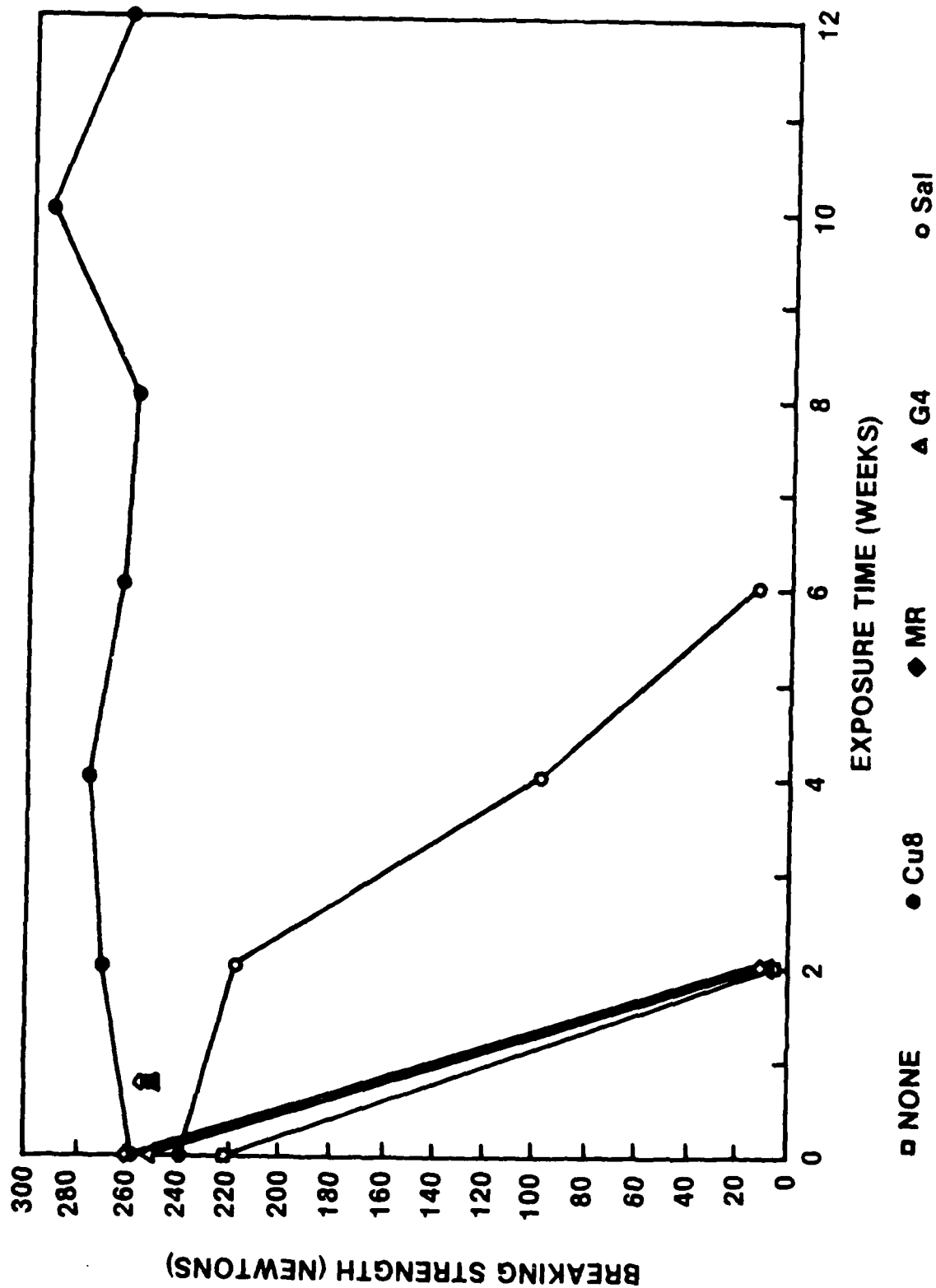


FIGURE 1. SOIL BURIAL DATA - UNLEACHED SPECIMENS.

MR=MOISTURE RESISTANT CONTROL

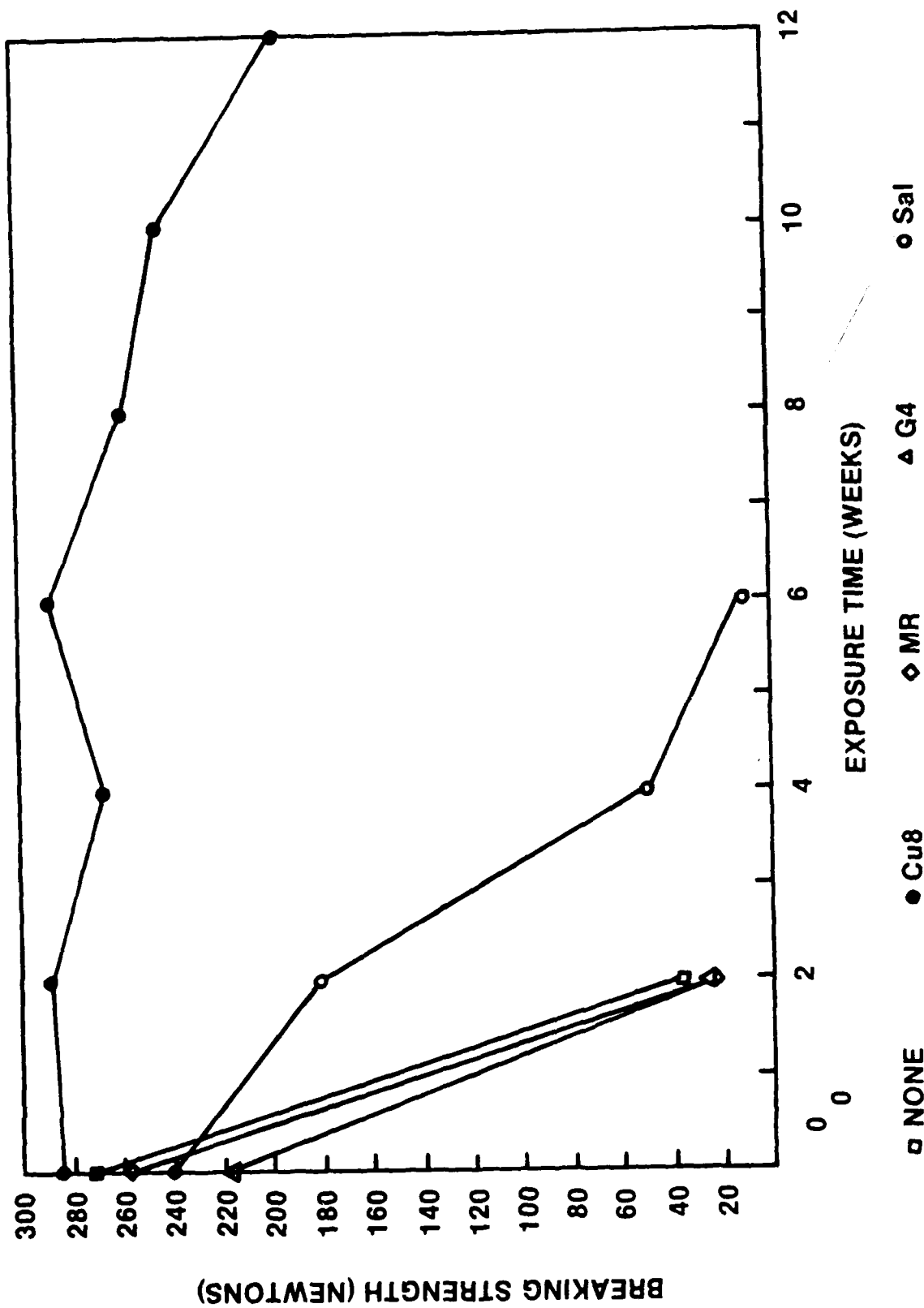


FIGURE 2. SOIL BURIAL DATA - LEACHED SPECIMENS.

DISCUSSION

The 3/16 inch (0.48 cm) thickness of the felt caused differential deposition of the fungicide with the center section being less concentrated than the outer sections - as much as 50% less in early trials. The application of heat or centrifugal tumbling to hasten drying increased the differential deposition of fungicide. Air drying without tumbling caused minimal fungicide gradient and was adopted as the standard application procedure for treating felt in this study.

Treated pieces were separately evaluated to ensure that both analytical and moisture resistance requirements were met. Moisture resistance requirements were easily met by all treatments. Analytical requirements, however, were more difficult to meet because of the tendency of the fungicide to migrate and deposit differentially during drying. Treated pieces were therefore evaluated by analyzing both the cross section and center section to make sure that minimal fungicide requirements were met throughout and also at the center of the felt pieces.

Analytical and moisture resistance data (Table 2) are from the treated pieces that met these requirements and were subsequently prepared for leaching and soil burial. Data from treated pieces not meeting these requirements were excluded from calculations and further evaluation.

Although all treated felt pieces were less wettable than the untreated control, the moisture resistant control was biodegraded as easily as the untreated control after soil exposure for 2 weeks. In this study the G-4 treatment offered no protection against biodegradation. The presence of sufficient G-4 throughout the felt analytically suggests that the G-4 formulation was a poor preparation and therefore ineffective. There was no evidence of incompatibility with the salicylanilide and Cu-8 treatments that were formulated and applied with the same base and solvent as the G-4 formulation.

The salicylanilide treatment provided less than four weeks of protection for unleached specimens in soil exposure and less than two weeks for leached specimens. The salicylanilide treatment therefore provided only marginal protection before greater than 10% loss in strength was found.

Cu-8, which was the trial fungicide in this study, provided the only durable protection of the three fungicides compared. Unleached Cu-8 treated specimens showed no biodegradation after soil exposure for 16 weeks. Leached Cu-8 treated specimens began to biodeteriorate after exposure for 12 weeks. As anticipated, the highly insoluble Cu-8 treatment was only slightly affected by leaching.

CONCLUSIONS

Of the three fungicides under comparison in this study, Cu-8 provided the only durable protection against biodeterioration of wool felt sheet. Salicylanilide and G-4 did not adequately protect the felt during soil burial tests. The laboratory data in this report suggest that Cu-8 should be included as an alternative fungicide in Military Specification MIL-F-2312E with the recommendation that it be first choice unless color or an incompatible property contraindicates.

The failure of G-4 to protect despite satisfactory chemical results points to the inadequacy of chemical methods to sometimes detect poor treatments. This is another instance where performance testing (in this case, soil burial) provided bottom line information otherwise unavailable. These data argue for the inclusion of soil burial in military specifications, where applicable, to assure adequate quality control.

This document reports research undertaken at the US Army Natick Research, Development and Engineering Center and has been assigned No. NATICK/TR-89011 in the series of reports approved for publication.

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